



(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
8 May 2003 (08/05/2003)

PCT

(10) International Publication Number
WO 03/037598 A2

(51) International Patent Classification: B29C 44/02, (74) Agent: WILSON, Kathryn, S. et al; Level 12, KPMG 44/12, 44/18, 35/08 Centre, 85 Alexander Street, Private Bag 3140, Hamilton 2001 (NZ).

(21) International Application Number: PCT/NZ02/000226

(22) International Filing Date: 29 October 2002 (29.10.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Date: 29 October 2001 (29.10.2001) NZ (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZM, ZW), European patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, ER, ES, FI, FR, GB, GR, IL, IT, LU, MC, NL, PT, SE, SK, TR, OA/P, patient (BE, BI, CF, CO, CI, CM, GA, GN, GG, GW, ML, MR, NE, SN, TD, TO).

(71) Inventors/Applicants (for US only): CHAPMAN, Thirby, James (NZ/NZ); 17 Hardwick Street, Sumner, Christchurch (NZ); ERRINGTON, John, George (NZ/NZ); 11 Paparoa Street, Paparoa, Christchurch (NZ); HASTINGS, Marlene (NZ/NZ); 16 Crofton Road, Harewood, Christchurch (NZ); HORNSEY, Aarya, Jane (NZ/NZ); 39A Scarborough Road, Sumner, Christchurch (NZ); QUINN, Philip, James (NZ/NZ); 259 Lutes Road, St Albans, Christchurch (NZ).

(75) Inventors/Applicants (for US only): CHAPMAN, Thirby, James (NZ/NZ); 17 Hardwick Street, Sumner, Christchurch (NZ); ERRINGTON, John, George (NZ/NZ); 11 Paparoa Street, Paparoa, Christchurch (NZ); HASTINGS, Marlene (NZ/NZ); 16 Crofton Road, Harewood, Christchurch (NZ); HORNSEY, Aarya, Jane (NZ/NZ); 39A Scarborough Road, Sumner, Christchurch (NZ); QUINN, Philip, James (NZ/NZ); 259 Lutes Road, St Albans, Christchurch (NZ).

Published:

— without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Note on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

IMPROVEMENTS IN AND RELATING TO BIO-DEGRADABLE FOAMED PRODUCTS

TECHNICAL FIELD

The present invention relates to improvements in the manufacture of bio-degradable foamed materials. More specifically the patent relates to an improved process of using microwaves to produce bio-degradable foamed shapes. The present invention further relates to the equipment and method used in producing foamed products.

BACKGROUND ART

The present invention builds on the invention disclosed in PCT/NZ01/00052. In this 5 patent application, a two stage process is described for producing a bio-degradable foamed product with improved packaging properties including resilience, compressibility and shock absorption. Definitions used in PCT/NZ01/00052 are included by reference herein.

The present invention builds on the invention disclosed in PCT/NZ01/00052. In this 10 patent application, a two stage process is described for producing a bio-degradable foamed product with improved packaging properties including resilience, compressibility and shock absorption. Definitions used in PCT/NZ01/00052 are included by reference herein.

The field of starch based bio-degradable foamed materials is widely discussed in the 15 prior art. A variety of products exist that attempt to produce bio-degradable foamed materials as discussed in PCT/NZ01/00052.

Extruded Starch Foams for Moulded Shapes

Patent US 5,750,824 (National Starch) utilises extrusion to produce foam panels. These panels are then laminated together to form thick sheets, which can be wire cut to varying 25 size shapes. This process has limitations due to the expensive capital equipment required for manufacturing. As a result of the expensive equipment, the method necessitates shipping 'air' as the product can only be made in central locations. In addition the shapes are either very 'limited' or 'obtuse' because they have to be cut out of sheets instead of moulded during the foaming process.

Another example, US 5,801,207 (Novafont) relates to foaming starch pieces, placing them in a bag or within layers of sheeting and moulding the pre-expanded

(54) **Title: IMPROVEMENTS IN AND RELATING TO BIO-DEGRADABLE FOAMED PRODUCTS**

(57) **Abstract:** The method provided produces a bio-degradable foamed material with qualities of uniformity of mechanical and physical properties throughout the product including a foam thickness of up to one metre and a finished foam surface suitable for packaging applications. The parameters for producing such a product are selected from a range of variables which includes wall thickness, mould material, use of a susceptor and the type and composition of a susceptor, the number and arrangement of magnetrons and mould shape. Complex shapes produced by the process are also disclosed.

peanuts into solid foam-in-place moulds. The limitations of this method are that the foamed peanuts used to make the moulds are very bulky and take up a lot of store space, and again increase expense through having to ship 'air' to the point of use instead of sending dense pellets that can be foamed at point of use. The method is also a complicated procedure for the end-user, as they have to fill and seal bags of foamed peanuts and then mould the bag to the product shape.

From the above it is hence useful to have a process that allows *in situ* foaming and further, that the equipment is relatively inexpensive and simple to use.

10 **Microwaved Starch Foams for Moulded Shapes**
Two main patents, WO9851466 (Ato-DI) and US 5,639,518 (NKCC), utilise dielectric heating in processing the starch based materials.

15 In WO9851466 (Ato-DI), the dielectric heating does not take into account the changing dielectric properties of the material as it heats, nor the relationship between the rheological properties (for example elasticity and viscosity) and the rate of heating. It further doesn't identify or address the detrimental effect of vapour condensation on the foam surface finish when such a process is used.

20 Patent US 5,639,518 (NKCC), utilises a number of different electromagnetic and electroconductive methods for producing foam bio-degradable shapes. They do not identify the importance of a rate of heating profile or specific rheology of the material being heated on the success of foaming thick walled bio-degradable shapes. They further do not identify or address the detrimental effect of vapour condensation on the foam surface finish when utilising microwave frequency.

A further patent, WO 02/02028, (Ato B.V.), details a process of steam heating taking 5 minutes to heat, under pressure, to the desired temperature range of 185°C. Such a long processing time reduces throughput significantly for a semi-continuous process.

30 In addition, the methods described above produce foams with varying consistency depending on the shape required and, often without the combination of uniform physical and mechanical properties. These properties include density, compressibility, resilience and shock absorption. All of these properties limit the product applications. It is

therefore desirable to have a method of processing that can produce a uniform product using equipment that is relatively inexpensive and simple to use.

Microwave Oven Designs

5 US 4,908,486 (Necartic Research Centre) describes a multiple magnetron microwave oven design where the oven is comprised of a cavity and at least one energy source. The main advantage disclosed of multiple energy sources is that the uniformity of drying is improved thus avoiding hot spots and cold spots inherent in some designs using only single energy sources. The oven is described as being useful for the drying of granular food crops including grain, rice, some fruits and beans. The apparatus does not however give consideration to use for foaming of materials, an object of the present invention. Further it does not consider the influence of adjusting the power density of the energy sources. The specification does not teach of processing multiple work pieces at any one time and further, does not address the use of moulds, shapes and objects other than granular materials.

10 It is hence useful to have an apparatus that addresses aspects such as variable energy density, and complex multiple work pieces.

20 Surface Coatings

In attempts to improve the surface texture or colour of microwaved products, susceptors have been considered in a number of applications, especially in domestic food applications. Susceptors are typically metallic films attached to microwave packages which are used in food applications to crisp or brown the food surface.

25 For foamed bio-degradable materials, where the goal is to produce a soft, smooth finish rather than the crisp texture desired in food applications, problems have been experienced surrounding vapour condensation at the interface between the mould wall and the foamed material. Although this problem is identified for example in US 5,965,080, (NKCC), this US patent refers to the problem of vapour breaking down an insulating layer on conductive moulds causing arcing, a processing problem specific to the use of conductive moulds, rather than the effect of vapour condensation on the surface finish of the foam.

4

A further patent, US 6,241,929 (Akopyan), recognises that uniformity is affected when the heat flow on the interface between the mould and the material is large and teaches that it is necessary for the material and the mould to have much the same dielectric properties. This patent, whilst describing a principle behind vapour condensation and its effect on uniformity, does not teach of specific processes and examples, particularly for bio-degradable foam applications.

It is therefore desirable to have a process utilising susceptors that also produces a uniform product with a smooth surface finish.

10 **10 Microwave Mould Designs**

US 5,965,080 (NKK) teaches of a method of foaming starch using conductive mould halves and an insulating section between. Both halves then have an alternating current applied thus heating and expanding the material. The importance of having vapour release sections is recognised as otherwise it is acknowledged that insulation breakdown occurs.

This method however has the problem that conductive moulds have a limited rate of heating range as arcing occurs with increased power densities. Uniformity is a further problem with this method in that fringe effects occur in corner areas. Further, complex shapes, which include a mix of thin and thick walled foam, are difficult to make using this method as the method is limited by arcing that occurs in thin walled areas.

Two alternative mould arrangements have been considered for expanded plastic materials.

15 **15 US4,298,324** (Isobox-Barbier) describes a device for moulding expanded plastic material. The device consists of a press, a mould body and resonant cavity combination. Mould surfaces in contact with material being moulded are formed from a resin containing carbon black, which has high dielectric losses, and the remaining portion of the mould body is made of a microwave transparent or transmissive material.

20 **20 US5,397,225** (Fuels) recognises the attributes of good dimensional accuracy and long serviceable lifetime for moulds to form latex foams with microwaves. Limitations of practicable wall thicknesses of typical microwave transparent materials are discussed as

are exposures to fluctuating temperatures. A new material based on polyphenylene ether with a passivated surface is described.

Whilst both methods describe useful alternatives, the limitations and constraints found from using bio-degradable materials are not considered.

5 **5 Mould Liners**

US5,508,498 (Invenetics) teaches of a utensil being a matrix material and a microwave absorptive material. The matrix is formed from silicone rubber with a ferrite based absorber material. The patent teaches only of use directed towards food applications and does not consider closed moulds or pressure changes that occur within the mould.

10 **10 US4,565,804** (CSEM) discusses use of a supporting body for analysing a product where the supporting body is comprised of a matrix material and a microwave absorptive material, evenly dispersed within the matrix material, and is characterised by a Curie temperature of 120-140°C. The invention is limited to a purpose of analysing thermally sensitive materials for volatile components and does not contemplate foaming of a low dielectric material like starch resin within an enclosed mould.

15 **15 US5,079,397** (Alcan) teaches of at least two regions of different lossiness in its susceptor materials. Examples of lossy substances suitable for inclusion in microwave susceptors are disclosed as well as techniques for application.

20 **20 None** of the above patents however account for use of a susceptor-type product with a closed mould for bio-degradable foams. In particular, they do not address the critical problems of susceptor and closed mould applications, being the prevention of condensation from vapour released, and the internal pressures that accumulate within a mould during starch based foaming processes.

25 **25 30 Thin Film**

A large number of patents refer to the use of thin films as a susceptor. For example, US5,019,681 (Pilkington) outlines prior art in the field of thin film susceptors where a thin layer such as polyester is used as the substrate with a thin metal film deposited on the substrate. US5,019,681 outlines further problems, specifically directed towards the

35

breakdown of the susceptor during heating leaving it only suitable for disposable single-use applications.

The prior art whilst helpful does not identify applications requiring and detailing the constraints necessary for successful bio-degradable foam applications. In particular, the prior art does not address the issues inherent to susceptors used in conjunction with closed moulds as described above. Namely, being the prevention of condensation from vapour released, while retaining a soft, smooth, surface finish, and the internal pressures that accumulate within a mould during starch based foaming processes.

10 *Other particulate options*

US5,294,763 (Minnesota Mining) describes particulate susceptors. Particulates susceptors can be divided into two categories; electrically continuous (e.g. carbon black) or electrically discontinuous (e.g. ferronagnetic particles).

15

Again the patent does not describe bio-degradable material forming applications and hence does not consider the particular problems associated with these materials.

It is an object of the current invention to overcome the limitations of the methods above.

20 It is a further object of the present invention to produce a foamed product with uniform physical and mechanical properties such as density, compressibility, resilience, shock absorption and surface finish by addressing the combination of problems with rate of heating, heating method and mould design in combination.

25 It is a further object of the present invention to produce a foamed product that is biodegradable and relatively inexpensive compared with previous methods.

It is an object of the present invention to address the foregoing problems or at least to 30 provide the public with a useful choice.

All references, including any patents or patent applications cited in this specification are hereby incorporated by reference. No admission is made that any reference constitutes prior art. The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinency of the cited documents. It will be clearly understood that, although a number of prior art publications

are referred to herein, this reference does not constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country.

5 It is acknowledged that the term 'comprise' may, under varying jurisdictions, be attributed with either an exclusive or an inclusive meaning. For the purpose of this specification, and unless otherwise noted, the term 'comprise' shall have an inclusive meaning - i.e. that it will be taken to mean an inclusion of not only the listed components it directly references, but also other non-specified components or elements. This rationale will also be used when the term 'comprised' or 'comprising' is used in relation to one or more steps in a method or process.

7 Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only.

15 DISCLOSURE OF INVENTION

For the purposes of this invention, a susceptor is defined as an article which contains microwave interactive material that absorbs microwave energy, and converts it into thermal energy. A susceptor may take many forms, including; a thin film; a liner; a surface coating on a mould. In a further alternative, the mould is the susceptor.

20 According to one aspect of the present invention there is provided a method of producing a bio-degradable foamed product wherein said product has the qualities of uniformity of mechanical and physical properties throughout the product;

a foam thickness of up to 1 metre;

25 a finished foam surface suitable for packaging applications

30 said qualities being achieved by the selection of parameters for moulding selected from the group including:

a selection of bio-degradable raw material processed into a form ready for foaming;

a selection of one or more magnetrons focused on a cavity, said magnetrons with a total power density of up to 10W/cm³;

a selection of one or more magnetrons by pre-determination of working volume, final product shape and mould shape;

a selection of one or more moulds to be heated at the same time;

8

a selection of mould wall thickness and shapes;
a selection of at least one mould material;
a selection of at least one susceptor or microwave interactive material;
and a combination of any of the above components.

5 In the preferred embodiment, the invention utilises domestic strength magnetrons thus restricting capital cost of the microwave machinery. Further a number of magnetrons are used in combination. It is understood by the applicant that this has the effect of improving the uniformity of the final product as well as reducing the apparatus expense.

10 By way of example a 15kW microwave generator has a cost over \$150,000 whereas a generator made up of 15 standard domestic 1kW magnetrons can be purchased for approximately \$25,000.

It is proven by the applicant that multiple work pieces can be used with the associated apparatus thus enabling the option of batch or semi-continuous processing of many pieces at once. The subsequent improvement in throughput is particularly advantageous and it is known by the applicant that the proposed method will allow for multiple work pieces.

15 Semi-continuous processing is also envisaged. One example includes that described in US 4,298,324 whereby a press, a resonant cavity and a mould structure are used. In an alternative a carrousel arrangement is used. In a further example considered by the applicant, a conveyor belt is used on which the work piece(s) travel along. The piece(s) are moved under the apparatus and the belt is raised forming a seal with the walls and ceiling of the microwave device. The seal avoids loss of microwave energy. As each piece finishes microwave processing the belt moves forward and the next set of work piece(s) enter the microwave. In an alternative embodiment, conveyors are used to load multiple cavity moulds into and out of a fixed cavity with a side door or doors, rather than the bottom floor sealing mechanism. It will be appreciated the other arrangements are also possible for semi-continuous operation.

20 In the above described method, the bio-degradable raw material is a bio-degradable polymer or additive selected from the group including: renewable natural resources and modifications of those; non-natural polymerisation of natural monomers or oligomers produced from natural resources; polymers obtained by biotechnological production and

25 30 35

other bio-degradable polymers such as polyvinyl alcohol (PVA) or polycaprolactone; and combinations thereof.

Other additives can also be included. Typically these additives are selected from a range of biodegradable plasticisers, nucleating agents, processing aids; and combinations thereof.

Further additives with an application dependent function can also be included such as flame retardants, fungus and mould inhibitors, strength adjusting additives, adhesion promoters, viscosity modifiers, fillers and rodent repellents.

The preferred method for preparing the material for foaming is by extrusion or similar heat and shear generating processes known in the art.

15 20 25 30 35

In the preferred embodiment, the processed material for foaming has a moisture content of 5 to 30% (w/w). The level of moisture has been found to be most preferable in the range from 15 to 22% (w/w).

In the preferred embodiment, the resulting product has similar mechanical properties to traditional materials. For example, polystyrene is a non bio-degradable material widely used for packaging. Preferred embodiments have comparable mechanical properties such as shock absorption and resilience.

According to a further aspect of the present invention, the base mould material is microwave or substantially microwave transparent. Examples include plastics, ceramics and glass. Preferably, plastics are selected from the group including: polyethylene (UHMWPE); acetyl; polysulfone (PSU); polypolyetherimide (ULTEM); polyetherketone (PEK); epoxy resins; polyphenylene ether; polypharylsulfone (PPS); and combinations thereof. Preferably, ceramics are selected from the group including gypsum (plaster of paris) and china clay.

In an alternative embodiment, plastic or ceramic mould materials are reinforced with a filler, microballoons, or glass fibres having low dielectric losses.

For the purposes of this specification, dielectric constant (relative permittivity) is associated with the electric field energy stored in the material. The dielectric constant is

10

PCTIN2002/000226

the ratio of the permittivity of a substance to the permittivity of free space. It is an expression of the extent to which a material concentrates electric flux.

Preferably base mould materials used have a dielectric constant of 0 to 10 at a frequency of 2.45 GHz and a loss factor of 0 to 0.1 at a frequency of 2.45 GHz. Most preferably materials are used with a dielectric constant of between 0 and 4 at a frequency 2.45 GHz and a loss factor of between 0 and 0.01 at a frequency of 2.45 GHz.

Preferably, moulds may include vent holes. These holes are positioned and sized according to the material and shape desired. Vent holes have the effect of allowing air and vapour to be released from the mould and hence tempering and/or removing pressure increases in the mould during processing.

According to a further aspect of the present invention, the mould also includes a susceptor (or is a susceptor itself) capable of absorbing and converting microwave energy into thermal energy while also transmitting sufficient microwave energy to the pellets.

It is understood by the applicant that the thermal energy generated by the mould elevates and maintains the mould surface temperature at a level that prevents the occurrence of condensation. Condensation in the mould has an adverse effect on the foam surface finish. By using a susceptor with appropriate conditions, foam with a smooth and resilient surface finish is achieved. The energy transmitted through the mould is at a level, which allows the required rate of heating of the pellets to be achieved.

In the applicant's experience, an elevated surface temperature also has the added advantage in that it aids in mould release. This is thought to be because the increased surface temperature reduces or eliminates condensation of steam. Steam typically breaks down the starch surface into a sticky substance thus making removal from moulds difficult.

Whilst the exact mechanism is not certain, it is the applicant's experience that maintaining or reducing the melt viscosity reduces the resistance to flow across the mould surface, resulting in an improved formation of the foam shape and hence finish.

35

11

It has been the applicant's experience that the temperature of the inner mould surface (susceptor) can be designed to reach steady state conditions in the desired temperature range. This results in the same temperature conditions being achieved during each moulding cycle, thus giving consistency between production runs.

5 Further, the mould surface temperature returns to a level where heat transfer from the mould to the pellets does not have an adverse effect on the pellets in the period between loading of the mould and microwave heating. Return to a temperature, which is below the point where significant vapour loss or burning of the pellets occurs, allows the mould to be reused.

10 Preferably the above elements are achieved by use of: a susceptor including; a thin film; a liner; or a surface coating on a mould. In a further alternative, the mould is the susceptor, with microwave interactive material dispersed throughout the mould material.

15 Preferably, the microwave interactive material in the susceptor is selected from the group including: electrically resistive or conductive materials, for example, a thin film of a metal or alloy such as aluminium; a resistive or semi conductive substance such as carbon black; graphite; silicon; silicon carbide; metal oxides; sulfides; ferrimagnetic materials such as iron or steel or ferrimagnetic alloys (stainless steel); ferrimagnetic materials such as acetal; and combinations thereof. Preferably, the susceptor is a liner which includes ferrite dispersed in silicone rubber or other resinous polymeric material

20 25 In an alternative embodiment, the mould itself is a susceptor impregnated with microwave interactive material selected from the group including: electrically resistive or conductive materials, for example, a thin film of a metal or alloy such as aluminium; a resistive or semi conductive substance such as carbon black; graphite; silicon; silicon carbide; metal oxides; sulfides; ferrimagnetic materials such as iron or steel or ferrimagnetic alloys (stainless steel); ferrimagnetic materials such as ferrites; a dielectric material such as acetal; and combinations thereof.

30 35 Preferably the mould surface temperature is greater than the melt temperature of the material being foamed and the temperature of the vapour given off during the process. It is the applicant's experience that in such an arrangement, foam with a soft, smooth surface finish and low abrasive characteristics is achieved.

Further embodiments of moulds include the ability to have thick foam shapes. A depth of up to 1 metre may be processed using the above apparatus combination giving uniform foaming and subsequent mechanical properties. It is the applicant's experience that the apparatus combination can be used to produce a wide variety of complex shapes only limited by the shape of the mould and the physical limitations of the microwave cavity size.

In a further aspect of the present invention, the material to be foamed and the mould are moved within the microwave field during foaming. It is understood by the applicant that this movement aids in improving the uniformity of the final foamed product.

In preferred embodiments, the microwave apparatus can be adjusted so that the rate of heating and the volume expansion of the material can be altered to obtain a uniform material. Preferred embodiments have the energy density variable from 0.001 to 10 W/cm³ and a rate of heating of 0.1-20°C per second temperature rise. More preferably the energy density is variable from 0.001 to 1 W/cm³ and the rate of heating is 5-10°C per second temperature rise.

20 A preferred frequency of operation for the microwave is from 100 MHz to 5 GHz. More preferably, one single frequency is used during processing. Most preferably, the frequency used is 2450 MHz.

A preferred power for the microwave apparatus is up to 100kW. It is understood by the applicant that the power requirement is however only limited by either the physical volume of the microwave cavity or the maximum power densities for a given volume.

Preferred embodiments of the microwave process may either utilise the microwave cycle described in PCT/NZ01/00052 or a single step cycle.

30 Pressure in the microwave cavity and/or mould in conjunction with rapid depressurisation can also be used to alter the final properties of the article such as foam density, shock absorption and finish. The parameters for such a process are considered in WO/02/20238 where pressures of up to 50 bar are considered.

Further controls to temperature and humidity can also be applied to the microwave cavity and/or mould during processing to vary the mechanical and surface finish characteristics of the material(s). Prior art suggests that temperatures in the range from 0°C to 250°C are useful.

5 In the present invention, the microwave equipment and/or process can be adjusted to give a finished foam density from 35 to 100 kg/m³. More preferably, this density ranges from 35 to 50 kg/m³. It has been found that this density gives desired physical and mechanical characteristics similar to that of non bio-degradable equivalent materials.

10 From the above method it is shown that a product can be produced that is bio-degradable, has similar mechanical properties to equivalent non-biodegradable materials and has a similar surface finish to alternative products. The process is relatively cheap in capital cost and labour cost compared to existing methods. Limitations of existing processes 15 including non-uniformity, adequate surface finish and low run batch operations, are resolved.

BRIEF DESCRIPTION OF DRAWINGS

Further aspects of the present invention will become apparent from the ensuing description which is given by way of example only and with reference to the accompanying drawings in which:

20 Figure 1 is an isometric view of rectangular block of foam;
Figure 2 is an isometric view of bottle mould foam and;
Figure 3 is an isometric view of a simple shaped foam (from a mould modified to fit a liner);
25 Figure 4 is an isometric view of a complex shaped foam;
Figure 5 is a graph showing the heating profiles used in Example 1;
Figure 6 is a graph showing the effect of surface temperatures on abrasive index as described in Example 2;
30 Figure 7 is a graph showing the temperature profile used in Example 6.

BEST MODES FOR CARRYING OUT THE INVENTION

In the preferred embodiment, the invention utilises a plurality of standard domestic magnetrons all concentrated on a cavity containing the material to be foamed and a microwave transparent mould.

5

In all of the following examples, a microwave consisting of twelve, 850-watt domestic magnetrons based around a power supply at 2450 megahertz is used for foaming the biodegradable material. The microwave cavity has a volume of approximately 0.4 m³ with a ceiling, walls, and floor according to known specifications.

10

Example 1

This example investigates the effect of rate of heating on the degree of foam formation and the density of the foam.

15 A mould is prepared for processing a shaped foam article as shown in Figure 3. The mould volume is approximately 1140 cm³, with a rectangular central section to the side of the product to be packaged. Multiple vent holes are present on the upper surface of the mould. The mould is made of ultra high molecular weight polyethylene (UHMWPE). No susceptor is used in this example.

The material for foaming consists of an extrudate, with a moisture content of 22% (w/w) and produced as per PCT/NZ01/00052 with a base material consisting of:

Material	Wt%
tapioca starch	86.75
polyvinyl alcohol	12
Lecithin	1
Magnesium silicate	0.25
TOTAL:	100.00

Table 1: Showing the raw material composition

25 125 grams of said material is placed into the mould and placed within the microwave cavity.

The samples are then microwaved at atmospheric pressure as follows:

Sample No.	Power Level	Microwave Time
1	1	260 seconds
2	3	80 seconds
3	6	44 seconds
4	12	24 seconds

Table 2: Showing the sample microwave conditions used

A graphical representation of the resulting heating profiles is shown in Figure 5.

5 The resultant foam has the following properties:

Sample No.	% Formation	Foam Density [kg/m ³]
1	50%	204
2	70%	144
3	90%	103
4	100%	91

Table 3: Showing % formation and foam density results

10 It can be seen from the above example that the higher rate of heating the better the foam formation achieved. It also shows that the higher the rate of heating, the lower the foam density achieved.

15 The higher rates of heating cause higher vapour pressures to be built up within the pellet, and hence a higher internal pressure within the mould. The higher internal pressure results in improved formation of the foam, as it is the internal pressure that forces the foam into the shape of the mould.

Example 2

This example investigates the effect of mould surface temperature on the surface finish and abrasive index of the foam, where results are compared with polystyrene and moulded pulp alternatives. The abrasive index represents the level of abrasion, which may occur between the foam and the product, which it is packaging.

Moulds to form a rectangular block as shown in Figure 1 are prepared for processing as follows:

- The mould volume for both moulds is approximately 1140 cm³, with a rectangular central section to site the product to be packaged. Multiple vent holes are present on the upper surface of the mould.
- Mould 1 is made of ultra high molecular weight polyethylene (UHMWPE) with a wall thickness of 25 mm.
- Mould 2 is made of acetal with a wall thickness of 15 mm.
- Silicone rubber and ferrite liners of varying compositions are used in each trial

Liner	1	2	3
Thickness:	1.6 mm	5.0 mm	5.0 mm
Weight % ferrite:	40%	40%	60%

Table 4: Showing the liner details

The material for foaming consists of an extrudate as described in Example 1.

Samples of 125 g of pellets are microwaved at atmospheric pressure on power level 12 as follows:

- Two trials were completed using UHMWPE moulds (mould 1) microwaved separately, with a microwave processing time of 24 seconds after which time the temperature and abrasive index was measured.
- One trial was completed using an acetal mould (mould 2) using a processing time of 46 seconds.
- Mould 1 (UHMWPE) was re-tested using liner 1 (40% ferrite @ 1.6mm) with a microwave processing time of 24 seconds.
- Mould 1 (UHMWPE) was re-tested using liner 2 (40% ferrite @ 5.0mm) with a microwave processing time of 24 seconds.
- Mould 1 (UHMWPE) was re-tested using liner 3 (60% ferrite @ 5.0mm) with a microwave processing time of 24 seconds.

The resultant foam also shown in the graph in Figure 6 has the following properties:

Sample	Packaging Material	Surface Temperature [°C]	Surface Finish	Abrasion Index
1	starch foam	29	Rough, brittle & pitted	10
2	starch foam	45	Rough, brittle & pitted	9.5
3	starch foam	60	Rough, brittle & pitted	9
4	starch foam	74	Rough, brittle & pitted	7
5	starch foam	80	Smooth, soft but resilient	3
6	starch foam	120	Smooth, soft but resilient	2
7	starch foam	160	Dry, weak & brittle	N/A
8	Polystyrene	N/A		2
9	moulded pulp	N/A		5

Table 5: Showing effect of surface temperature on surface finish and abrasive index

Abrasive Index Scale: 0 → 10 Highly abrasive; 10 Low Abrasion: 0 (polystyrene and moulded pulp properties are given by way of reference).

5

From the above results it can be seen that by elevating the surface temperature of the mould, the quality of the surface finish of the foam is improved. This is evident in both the recorded observations and the abrasion index measurement. It can also be seen that the surface finish achieved on the starch foam is comparable with that of polystyrene and superior to that of moulded pulp packaging.

Steam, given off during the process, condenses on the mould walls and the condensate causes the cellular structure of outer surface of the foam to collapse. It also causes pitting and the formation of a hard, brittle and abrasive surface finish. If the temperature of the inner mould surface is elevated, condensation of the steam is prevented and the resulting foam surface finish is highly improved.

Example 3

This example investigates the effect of elevation and control of the mould surface temperature on the degree of foam formation.

The UHMWPE mould described in Example 2 and the liners described below, were used in this example to complete a total of nine trials.

Liner	1	2	3
Thickness:	1.6 mm	2.5mm	5.0 mm
Weight % ferrites:	40%	40%	40%

Table 6: Showing the mould details

- Using an UHMWPE mould and liner 1, three separate loads of 125 g of pellets were microwaved on power level 12 for 24 seconds.
- Using an UHMWPE mould and liner 2, three separate loads of 125g, 135g and 145g of pellets were microwaved on power level 12 for 24 seconds.
- Using an UHMWPE mould and liner 3, three separate loads of 125g, 135g and 145g of pellets were microwaved on power level 12 for 24 seconds.

10 The resultant foam had the following properties:

In this example, the material for foaming consists of an extrudate, as described in Example 1. The microwave geometry is maintained the same throughout the experiment as that of earlier examples. Four different shaped moulds are used as follows:

Mould	1	2	3	4
Mould Name:	Rectangular Block (as shown in Figure 1)	Bottle Mould (as shown in Figure 2)	Simple End Cap (as shown in Figure 3)	Complex End Cap (as shown in Figure 4)
Material:	UHMWPE	UHMWPE	UHMWPE	UHMWPE
Wall thickness:	25 mm	25 mm	25 mm	25 mm
Volume:	0.00145 m ³	0.001114 m ³	0.00127 m ³	0.00184 m ³

Table 8: Showing the mould details

The following trials were then completed where each mould was placed into the microwave cavity individually and treated as follows:

Mould	Pellet Load	Processing Time
1 (Rectangular)	140 g	30 seconds
2 (Bottle Mould)	115g	24 seconds
3 (Simple End Cap)	125g	24 seconds
4 (Complex End Cap)	220g	38 seconds

Table 9: Showing the mould, the amount of raw material used and the microwave conditions

Sample	Temp [°C]	Density [kg/m ³]	Formation
Liner 1, Sample 1	60	105	100%
Liner 1, Sample 2	60	95	80%
Liner 1, Sample 3	60	85	70%
Liner 2, Sample 4	80	105	100%
Liner 2, Sample 5	80	95	90%
Liner 2, Sample 6	80	85	80%
Liner 3, Sample 7	120	105	100%
Liner 3, Sample 8	120	95	100%
Liner 4, Sample 9	120	85	100%

Table 7: Showing improved degree of formation at lower densities through elevation of surface temperature

It can be seen from this example that a higher surface temperature results in full foam formation at a lower density than occurs with a lower surface temperature.

Example 4

This example demonstrates how foam shapes of both simple and complex geometries can be processed using the same microwave configuration.

Trial	Power Level	Process Time	Mass Pellets	Mass Foam	% Formed	Density
1	12	30 sec	153 g	132 g	100%	91 g/L
2	12	24 sec	110 g	94 g	100%	82.5 g/L
3	12	24 sec	125 g	106 g	100%	83.5 g/L
4	12	38 sec	220 g	186 g	100%	101 g/L

Table 10: Showing the success of moulding different shapes using the same microwave generator equipment

The above trial shows that a wide variety of shapes can be processed giving uniform properties via the same microwave equipment thus reducing costs associated with microwave equipment modifications and labour costs in manufacturing such pieces.

5 Example 5
This example investigates whether or not various silicone/ferrite liners come to steady state temperature and if so, at what steady state temperature is a good surface finish achieved?

10 In this example, the material for foaming consists of an extrudate, as described in Example 1.
A mould is used as described in Example 1 (UHMWPE).

15 Three different types of silicone/ferrite liner are trialed as follows:

Liner	1	2	3
Thickness:	1.6 mm	2.5mm	5.0mm
Weight % Ferrite:	40%	40%	40%

Table 11: Showing the Liner details

15 A sample of 125g of starch pellets was microwaved in the UHMWPE mould fitted with liner 1 at power level 12 and with a microwave processing time of 30 seconds. The process was repeated for liners 2 and 3.

15 The resulting foamed products gave the following properties:

Trial	Steady State Surface Temperature [C]	Foam Surface Finish	Number of Runs Required to Achieve Steady State	Total Number of Runs
Liner 1	60	Hard pitted surface	4	20
Liner 2	120	Smooth soft surface	4	20
Liner 3	190	Thermal degradation of foam, foam surface dry and brittle	4	20

Table 12: Showing the effect of surface temperature on foam finish

As in example 2, elevating the surface temperature of the mould is found to improve the surface finish of the foam. This example also shows that a ferrite/silicone liner can be designed so that it comes to steady state in the desired temperature range. Achieving steady state is of major significance as it allows the mould to be used repeatedly without delay in a production environment. If steady state were not achieved, variation of product quality would be experienced and thermal runaway would be likely.

10 **Example 6**
This example illustrates how a thin film metal susceptor can be used to generate sufficient surface heating to prevent condensation and improve the surface finish on the foam.

15 In this example, the material for foaming consists of an extrudate, as described in Example 1.

An UHMWPE mould of volume 1140 cm³ is used, laminated with a polyethylene terephthalate aluminium (Al/PET) film. The aluminium thickness is approximately 0.02 20 microns.

A sample of 125g of starch pellets were placed in the lined mould and microwaved at power level 12 with a microwave processing time of 24 seconds. The temperature profile for the aluminium/PET film is shown in Figure 7.

25 The resulting foamed product gave a surface result with a smooth, soft, but resilient surface. A comparative mould without an aluminium/PET film yields foam with a rough, brittle and pitted surface.

30 The example shows that the thin film aluminium heats when exposed to microwave energy as the result of an IR (Ohmic) heating mechanism. This heating generates a surface temperature sufficient to prevent condensation and yield foam with an improved surface finish. Films that generate a surface temperature above this range (180°C) result in browning/burning of the foam surface.

35

Example 7

This example investigates the effect of matching the mould surface temperature to the melt temperature of the material. It is known that if no temperature gradient exists then no net transfer of energy can occur.

5

In this example, the material for foaming consists of an extrudate, as described in Example 1. A mould is used as described in Example 1 (UHMWPE).

A sample of 125g of starch pellets was microwaved in the UHMWPE mould at a microwave power level of 12 with a microwave processing time of 24 seconds. A similar experiment was also completed whereby the mould surface temperature was less than the melt temperature.

The results were as follows:

15

Example	Temperature	Foam surface
Figure 1	T _{surface} > T _{melt} & T _{vapour}	Soft smooth surface finish
Figure 2	T _{surface} < T _{melt} & T _{vapour}	Hard, brittle, pitted surface finish

Table 13: showing the effect of surface temperature against that of the melt temperature

It can be seen from the above example that where the surface temperature is less than that of the melt temperature, a poor foam surface finish is achieved.

20

From the examples it can be seen that a variety of moulds and options for altering the surface finish can be used as required. In particular mould surface temperature modifiers particularly aid finish. The process produces a product with comparable qualities to alternative non-biodegradable products such as polystyrene. Further, the process cost is minimised by utilising standard domestic magnetrons rather than very expensive high power magnetrons.

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope thereof as defined in the appended claims.

30

WHAT WE CLAIM IS:

1. A method of producing a bio-degradable foamed product wherein said product has the qualities of uniformity of mechanical and physical properties throughout the product;
 - 5 a foam thickness of up to 1 metre;
 - a finished foam surface suitable for packaging applications;
 - said qualities being achieved by the selection of parameters for moulding selected from the group including:
 - 10 a selection of bio-degradable raw material processed into a form ready for foaming;
 - a selection of one or more magnetrons focused on a cavity with a total power density of up to 10W/cm³;
 - a selection of one or more magnetrons by pre-determination of working volume, final product shape and mould shape;
 - a selection of one or more moulds to be heated at the same time;
 - a selection of mould wall thickness and shapes;
 - a selection of at least one mould material;
 - a selection of at least one susceptor on microwave interactive material;
 - 15 and a combination of any of the above components.
2. A method of producing a bio-degradable foamed product as claimed in claim 1 which utilises domestic strength magnetrons.
3. A method of producing a bio-degradable foamed product as claimed in either claim 1 or claim 2 whereby semi continuous processing is completed by:
 - 20 (i) the piece or pieces on a conveyor belt are moved under a microwave apparatus;
 - (ii) the belt forms a microwave seal with the walls and ceiling of the microwave apparatus and microwave processing commences;
 - (iii) as the piece or pieces finish microwave processing, the belt moves forward and the next piece or pieces enter the microwave apparatus.
4. A method of producing a bio-degradable foamed product as claimed in either claim 1 or claim 2 whereby semi continuous processing is completed by:

(i) the pieces or pieces on a conveyor belt are moved into a microwave apparatus cavity;

(ii) a door seals off the apparatus cavity and microwave processing occurs;

(iii) as the piece or pieces finish microwave processing, the door is opened and the belt moves forward and the next piece or pieces enter the microwave apparatus cavity.

5. A method of producing a bio-degradable foamed product as claimed in any one of the above claims, wherein the bio-degradable raw material is a bio-degradable polymer or additive selected from the group including: renewable natural resources and modifications of those; non-natural polymerisation of natural monomers or oligomers produced from natural resources; polymers obtained by biotechnological production and other bio-degradable polymers such as polyvinyl alcohol (PVA) or polycaprolactone; and combinations thereof.

6. A method of producing a bio-degradable foamed product as claimed in claim 5, wherein the bio-degradable raw material includes additives selected from the group including: biodegradable plasticisers; nucleating agents; processing aids; and combinations thereof.

7. A method of producing a bio-degradable foamed product as claimed in claim 5 or 6 wherein the method for preparation of the foaming material from the raw material is by a heat and shear generating process.

8. A method of producing a bio-degradable foamed product as claimed in claim 7 wherein the method for preparation of the foaming material is by extrusion.

9. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the bio-degradable material for foaming has a moisture content of 5 to 30% (w/w).

10. A method of producing a bio-degradable foamed product as claimed in claim 9 wherein the bio-degradable material for foaming has a moisture content of 15 to 22% (w/w).

11. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the base mould material is substantially microwave transparent.

12. A method of producing a bio-degradable foamed product as claimed in any one of the above claims, wherein the base mould materials are selected from the group including: plastics; ceramics; glass; and combinations thereof.

13. A method of producing a bio-degradable foamed product as claimed in claim 12 wherein the plastics are selected from the group including: polyethylene (UHMWPE); acetate; polyulfone (PSU); polypolyetherimide (ULTEM); Polyetherketone (PEEK); epoxy resins; polyphenylene ether; polyphenylsulfone (PPSU); and combinations thereof.

14. A method of producing a bio-degradable foamed product as claimed in claim 12 wherein ceramics are selected from the group including gypsum (Plaster of paris) and china clay.

15. A method of producing a bio-degradable foamed product as claimed in any one of the above claims, wherein the mould material is reinforced with a filler, microballoons, or glass fibres having low dielectric losses.

16. A method of producing a bio-degradable foamed product as claimed in any one of claims 11 to 15 wherein the base mould material used has a dielectric constant of 0 to 10 at a frequency of 2.45 GHz and a loss factor of 0 to 0.1 at a frequency of 2.45 GHz.

17. A method of producing a bio-degradable foamed product as claimed in claim 16 wherein the base mould material used has a dielectric constant of between 0 and 4 at a frequency of 2.45 GHz and a loss factor of between 0 and 0.01 at a frequency of 2.45 GHz.

18. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein moulds include vent holes.

19. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the susceptor including microwave interactive material is selected from the group including: a thin film; a liner; a surface coating.
20. A method of producing a bio-degradable foamed product as claimed in any one of claims 1 to 10 and 12 to 19, wherein the mould is the susceptor and microwave interactive material is dispersed throughout the mould material.
21. A method of producing a bio-degradable foamed product as claimed in claim 19 or 20 wherein the microwave interactive material is selected from the group including: electrically resistive or conductive materials, for example, a thin film of a metal or alloy such as aluminum; a resistive or semi conductive substance such as carbon black; graphite; silicon carbide; metal oxides; sulfides; ferromagnetic materials; ferrimagnetic materials; a dielectric material; and combinations thereof.
22. A method of producing a bio-degradable foamed product as claimed in claim 19 wherein the susceptor is a liner with ferrite dispersed in silicone rubber or other resinous polymeric material.
23. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the susceptor elevates the surface temperature of the mould from 50°C to 190°C.
24. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the selected susceptor reaches steady state operating conditions, where the maximum surface temperature is from 50°C to 190°C.
25. A method of producing a bio-degradable foamed product as claimed in claim 24 wherein the steady state of operating conditions, where the maximum surface temperature is from 80°C to 140°C.
26. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the surface temperature of the mould is greater than the melt temperature of the material being foamed and the temperature of any vapour given off during the process.

27. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the mould and material to be foamed are moved within the microwave field during foaming.
28. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the microwave apparatus energy density is set at or varied from 0.001 to 10 W/cm² during processing.
29. A method of producing a bio-degradable foamed product as claimed in claim 28 wherein the energy density is set at or varied from 0.001 to 1 W/cm² during processing.
30. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the rate of heating within the material being foamed is set at or varied from 0.1-20°C per second temperature rise during processing.
31. A method of producing a bio-degradable foamed product as claimed in claim 30 wherein the rate of heating is set at or varied from 5-10°C per second temperature rise.
32. A method of producing a bio-degradable foamed product as claimed in any one of the previous claims wherein the frequency of operation for the microwave is selected from any one frequency, or varied in frequency during processing, from 100 MHz to 5 GHz.
33. A method of producing a bio-degradable foamed product as claimed in claim 32 wherein the frequency used is approximately 2450MHz.
34. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the power for the microwave apparatus is up to 100kW.
35. A method of producing a bio-degradable foamed product as claimed in any one of the above claims wherein the power for the microwave apparatus is up to 100kW. the energy density remain the same for different shaped foamed objects.

36. A method of producing a bio-degradable foamed product as claimed in any one of the claims above wherein the microwave equipment is adjusted to give a finished foam density from 35 to 100 kg/m³.

37. A method of producing a bio-degradable foamed product as claimed in claim 36 wherein the finished foam density is from 35 to 50 kg/m³.

38. A foamed product produced by the method as claimed in any one of claims 1 to 37.

39. A method of producing a bio-degradable foamed product substantially as claimed in any one of claims 1 to 37, as hereinbefore described and with reference to the accompanying drawings and examples.

40. A foamed product substantially as claimed in claim 38, as hereinbefore described and with reference to the accompanying drawings and examples.

1/4

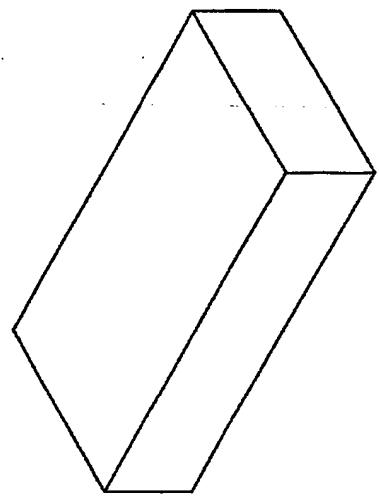


Figure 1

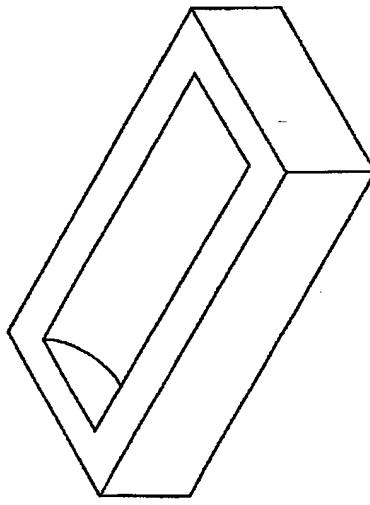


Figure 2

2/4

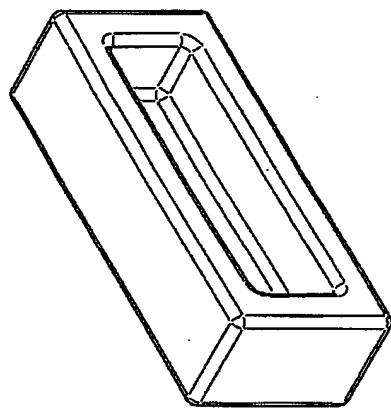


Figure 3

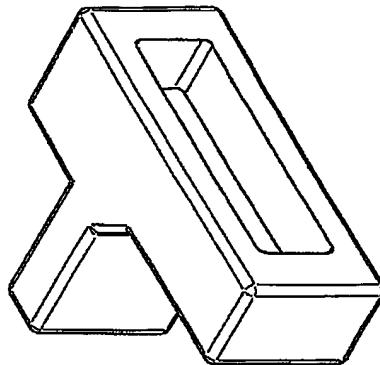


Figure 4

3/4

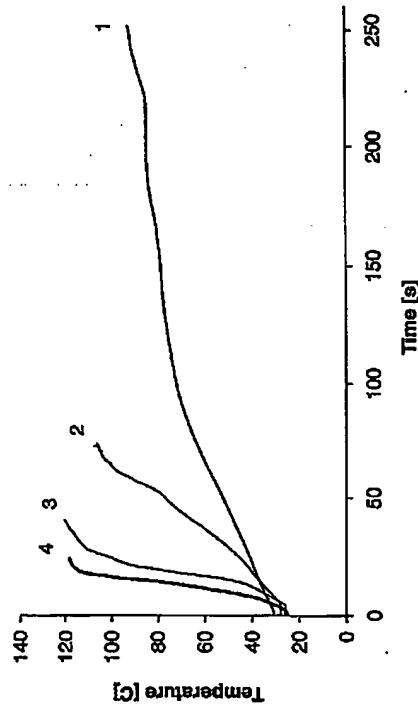


Figure 5

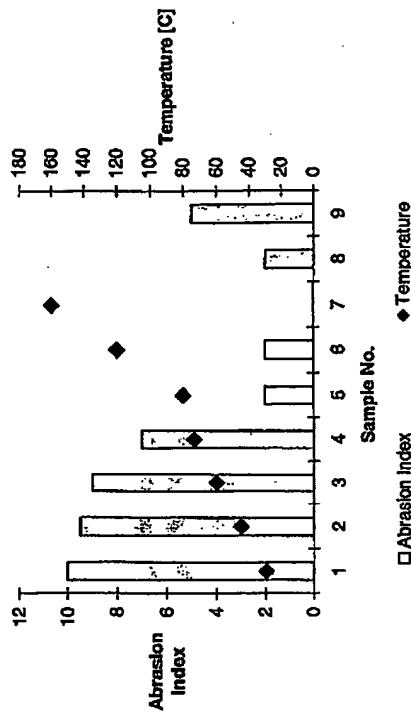


Figure 6

4/4

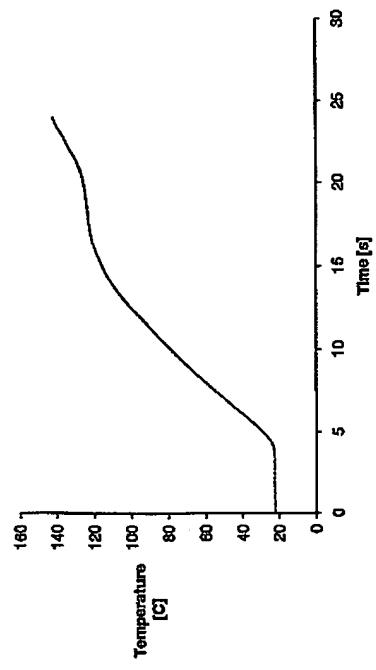


Figure 7

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.